

FORM PTO-1390 (Modified)
(REV 11-2000)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371

217771US0PCT

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR

10/030424

INTERNATIONAL APPLICATION NO.
PCT/JP00/04571INTERNATIONAL FILING DATE
07 JULY 2000PRIORITY DATE CLAIMED
13 JULY 1999 (EARLIEST)

TITLE OF INVENTION

POLISHING LIQUID COMPOSITION

APPLICANT(S) FOR DO/EO/US

Yasuhiro YONEDA, et al.

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (24) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371 (c) (2))
 - a. ☐ is attached hereto (required only if not communicated by the International Bureau).
 - b. ☒ has been communicated by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
 - a. ☐ is attached hereto.
 - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))
 - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ have been communicated by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
10. ☐ An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).
11. ☒ A copy of the International Preliminary Examination Report (PCT/IPEA/409).
12. ☒ A copy of the International Search Report (PCT/ISA/210).

Items 13 to 20 below concern document(s) or information included:

13. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
14. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
15. ☒ A **FIRST** preliminary amendment.
16. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
17. ☐ A substitute specification.
18. ☐ A change of power of attorney and/or address letter.
19. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
20. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
21. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
22. ☐ Certificate of Mailing by Express Mail
23. ☒ Other items or information:

Notice of Priority / PTO-1449

10030424 011002

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR 1.101) 10/030424		INTERNATIONAL APPLICATION NO. PCT/JP00/04571		ATTORNEY'S DOCKET NUMBER 217771US0PCT	
---	--	--	--	---	--

24. The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)) :

<input type="checkbox"/> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO	\$1040.00
<input checked="" type="checkbox"/> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO	\$890.00
<input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO	\$740.00
<input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4)	\$710.00
<input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4)	\$100.00

ENTER APPROPRIATE BASIC FEE AMOUNT =

Surcharge of **\$130.00** for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492 (e)).

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	
Total claims	9 - 20 =	0	x \$18.00	\$0.00
Independent claims	3 - 3 =	0	x \$84.00	\$0.00
Multiple Dependent Claims (check if applicable) <input type="checkbox"/>				\$0.00
TOTAL OF ABOVE CALCULATIONS =				\$890.00
<input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.				\$0.00
SUBTOTAL =				\$890.00
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492 (f)).				\$0.00
TOTAL NATIONAL FEE =				\$890.00
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) (check if applicable) <input type="checkbox"/>				\$0.00
TOTAL FEES ENCLOSED =				\$890.00
				Amount to be: refunded \$
				charged \$

CALCULATIONS PTO USE ONLY

a. ☒ A check in the amount of \$890.00 to cover the above fees is enclosed.


b. ☐ Please charge my Deposit Account No. _____ in the amount of _____ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 15-0030 A duplicate copy of this sheet is enclosed.

d. ☐ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. **Credit card information should not be included on this form.** Provide credit card information and authorization on PTO-2038.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:



22850

Surinder Sachar

Registration No. 34,423

(703) 413-3000

Surinder Sachar

SIGNATURE

Norman F. Oblon

NAME

24,618

REGISTRATION NUMBER

 Jan 10 2002

DATE

217771US-0 PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :
YASUHIRO YONEDA ET AL : ATTN: APPLICATION DIVISION
SERIAL NO: NEW U.S. APPLICATION :
(Based on PCT/JP00/04571)
FILED: HEREWITH :
FOR: POLISHING LIQUID COMPOSITION :

PRELIMINARY AMENDMENT

ASSISTANT COMMISSIONER FOR PATENTS
WASHINGTON, D.C. 20231

SIR:

Prior to examination on the merits, please amend the above-identified application as follows:

IN THE CLAIMS

Please cancel Claim 8.

Please amend the claims as shown on the marked-up copy following this amendment to read as follows:

7. (Amended) The polishing liquid composition according to Claim 1, further comprising an oxidizing agent, an abrasive or a mixture thereof.

9. (Amended) A process for polishing a semiconductor substrate, comprising polishing a surface to be polished comprising an insulating layer and a metal layer using the polishing liquid composition of Claim 1, thereby smoothening the semiconductor substrate.

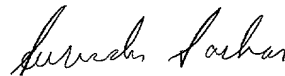
10. (Amended) A process for manufacturing a semiconductor substrate comprising polishing a surface to be polished comprising an insulating layer and a metal layer using the polishing liquid composition of Claim 1, thereby smoothening the semiconductor substrate.

REMARKS

Claims 1-7 and 9-10 are active in the present application. Claim 8 has been canceled. Claims 7 and 9-10 have been amended to remove multiple dependencies. Claim 7 has been amended to include further components of the polishing liquid composition. Amended Claim 7 is supported by the specification on page 5, lines 1-3. No new matter is added. An action on the merits and allowance of claims is solicited.

Respectfully submitted,

OBLON, SPIVAK, McCLELLAND,
MAIER & NEUSTADT, P.C.



Norman F. Oblon
Attorney of Record
Registration No. 24,618

Stefan U. Koschmieder, Ph.D.
Registration No. 50,238



22850

(703) 413-3000
Fax #: (703)413-2220
NFO/SUK/sjh
I:\atty\SUKOS\217771US-PR.WPD

Surinder Sachar
Registration No. 34,423

Marked-Up Copy

Serial No:

Amendment Filed on:

1-10-2002

IN THE CLAIMS

7. (Amended) The polishing liquid composition according to [any one of claims 1 to 5] Claim 1, further comprising an oxidizing agent, an abrasive or a mixture thereof.

8. (Canceled).

9. (Amended) A process for polishing a semiconductor substrate, comprising polishing a surface to be polished comprising an insulating layer and a metal layer using the polishing liquid composition of [any one of claims 1 to 8] Claim 1, thereby smoothening the semiconductor substrate.

10. (Amended) A process for manufacturing a semiconductor substrate comprising polishing a surface to be polished comprising an insulating layer and a metal layer using the polishing liquid composition of [any one of claims 1 to 8] Claim 1, thereby smoothening the semiconductor substrate.

DESCRIPTIONPOLISHING LIQUID COMPOSITION5 TECHNICAL FIELD

The present invention relates to a polishing liquid composition for polishing a surface to be polished comprising an insulating layer and a metal layer. More specifically, the present invention relates to a polishing liquid composition which is applicable as a means of forming embedded metal interconnection on a semiconductor substrate, a process for polishing, and a process for manufacturing a semiconductor substrate.

BACKGROUND ART

In the process for manufacturing a semiconductor device, comprising the steps of forming interconnection-shaped recesses on a surface of the insulating film on a semiconductor substrate, sedimenting a metal film made of copper or the like on the insulating film having the recesses, and subjecting the metal film to polishing treatment by a polishing device and a polishing liquid, thereby allowing the metal layer to remain only in the recesses to form a metal interconnection layer, wherein Metal Chemical Mechanical Polishing (hereinafter simply referred to as "metal CMP") is employed for the process of polishing.

However, in the metal CMP, there arise grooves so-called dishing on the metal interconnection layer in the recesses of the insulating film, so that the cross-sectional area of the metal interconnection layer is reduced,

thereby causing an increase in electric resistivity. This dishing is caused by more excessive polishing or etching of the surface of the metal interconnection layer than that of the insulating film surface by the polishing liquid composition. Especially copper, one of the main metal interconnection, has defects of being excessively etched by the polishing liquid composition, so that the dishing is likely to be caused.

Therefore, there has been desired a polishing liquid composition free from defects such as dishing in the metal layer during the formation of interconnection, with retaining an etching action for polishing the metal film on the insulating film.

As a conventional polishing liquid, for instance, Japanese Patent Laid-Open Nos. Hei 8-83780 and Hei 11-21546 each discloses a polishing liquid comprising benzotriazole or a derivative thereof as a protective film-forming agent for the metal surface in order to prevent the dishing. Since the formed protective film is rigid, when the metal layer is polished in the metal CMP, the polishing speed would become insufficient. In addition, Japanese Patent Laid-Open No. Hei 11-116942 discloses a composition for polishing, comprising a compound having 1 to 10 alcoholic hydroxyl groups, or a nitrogen-containing basic compound having 1 to 10 alcoholic hydroxyl groups. This composition for polishing has a purpose of reducing particles deposited on a wafer surface in final polishing of the semiconductor wafer, so that the problems to be solved are different.

In addition, Japanese Patent Laid-Open No. Hei 10-44047 discloses a polishing liquid comprising an aqueous medium, an abrasive, an oxidizing agent, and an organic acid. However, since the etching action is too strong,

the prevention for dishing would be insufficient. Further, Japanese Patent Laid-Open No. Hei 11-195628 discloses a process for polishing in which a polishing liquid is used in combination with ammonium polyacrylate as a substance for suppressing oxidation and etching. However, in the metal CMP in which a metal layer made of copper or the like is polished, there arises surface roughening of the copper surface caused by ammonium polyacrylate.

An object of the present invention is to provide a polishing liquid composition capable of maintaining a polishing speed of a metal film, suppressing an etching speed, and having an excellent prevention effect such as dishing of the metal interconnection layer, in a surface to be polished comprising an insulating layer and a metal layer; a process for polishing; and a process for manufacturing a semiconductor substrate.

These objects and other objects of the present invention will be apparent from the following description.

DISCLOSURE OF INVENTION

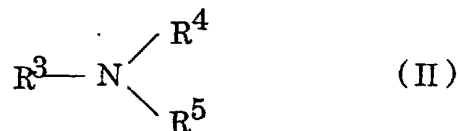
Specifically, the present invention relates to:

[1] a polishing liquid composition for polishing a surface to be polished comprising an insulating layer and a metal layer, the polishing liquid composition comprising a compound having a structure in which each of two or more adjacent carbon atoms has a hydroxyl group in a molecule, and water (hereinafter also referred to "the polishing liquid composition 1-1");

[2] a polishing liquid composition for polishing a surface to be polished comprising an insulating layer and a metal layer, the polishing liquid

composition comprising an aliphatic carboxylic acid having 7 to 24 carbon atoms and/or a salt thereof, an etching agent, and water (hereinafter also referred to "the polishing liquid composition 1-2");

[3] a polishing liquid composition for polishing a surface to be polished comprising an insulating layer and a metal layer, the polishing liquid composition comprising an amine compound represented by the following general formula (II):



wherein R³ is a linear or branched alkyl group having 4 to 18 carbon atoms, a linear or branched alkenyl group having 4 to 18 carbon atoms, an aryl group having 6 to 18 carbon atoms, and an aralkyl group having 7 to 18 carbon atoms; each of R⁴ and R⁵, which may be identical or different, is hydrogen atom, a linear alkyl group having 1 to 8 carbon atoms or a branched alkyl group having 3 to 8 carbon atoms, or a group represented by H-(OR⁶)_Z-, wherein R⁶ is a linear alkylene group having 1 to 3 carbon atoms, or a branched alkylene group having 3 carbon atoms; and Z is a number of 1 to 20,

and/or a salt thereof, an etching agent, and water (hereinafter also referred to "the polishing liquid composition 1-3");

[4] the polishing liquid composition according to any one of items [1] to [3] above, further comprising an oxidizing agent (hereinafter also referred to "the polishing liquid composition 2");

[5] the polishing liquid composition according to any one of items [1] to [4] above, further comprising an abrasive (hereinafter also referred to "the polishing liquid composition 3");

[6] a process for polishing a semiconductor substrate, comprising
5 polishing a surface to be polished comprising an insulating layer and a metal layer using the polishing liquid composition of any one of items [1] to [5], thereby smoothening the semiconductor substrate; and

[7] a process for manufacturing a semiconductor substrate comprising
10 polishing a surface to be polished comprising an insulating layer and a metal layer using the polishing liquid composition of any one of items [1] to [5], thereby smoothening the semiconductor substrate.

BEST MODE FOR CARRYING OUT THE INVENTION

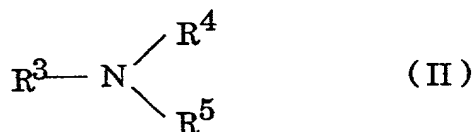
As mentioned above, the polishing liquid composition of the present
15 invention is a polishing liquid composition for polishing a surface to be polished comprising an insulating layer and a metal layer, and has the following three embodiments.

Embodiment 1: A polishing liquid composition comprising a compound
20 having a structure in which each of two or more adjacent carbon atoms has a hydroxyl group in a molecule, and water.

Embodiment 2: A polishing liquid composition comprising an aliphatic carboxylic acid having 7 to 24 carbon atoms and/or a salt thereof, an etching agent and water.

25 Embodiment 3: A polishing liquid composition comprising an amine

compound represented by the following general formula (II):



wherein R³ is a linear or branched alkyl group having 4 to 18 carbon atoms, a linear or branched alkenyl group having 4 to 18 carbon atoms, an aryl group having 6 to 18 carbon atoms, and an aralkyl group having 7 to 18 carbon atoms; and each of R⁴ and R⁵, which may be identical or different, is hydrogen atom, a linear alkyl group having 1 to 8 carbon atoms or a branched alkyl group having 3 to 8 carbon atoms, or a group represented by H-(OR⁶)_Z-, wherein R⁶ is a linear alkylene group having 1 to 3 carbon atoms, or a branched alkylene group having 3 carbon atoms; and Z is a number of 1 to 20, and/or a salt thereof, an etching agent, and water.

Embodiment 1

In this embodiment, one of the great features resides in the use of a compound having a structure in which each of two or more adjacent carbon atoms has a hydroxyl group in a molecule (hereinafter simply referred to as "hydroxyl group-containing compound"). By the use of the polishing liquid composition comprising the hydroxyl group-containing compound, the polishing speed can be maintained, and excessive etching of a metal film in the metal layer can be prevented, so that there is exhibited an excellent effect that a polishing surface without defects such as dishing can be

obtained.

In the hydroxyl group-containing compound, from the viewpoint of maintaining the polishing speed and suppressing dishing, the number of adjacent carbon atoms having a hydroxyl group in a molecule is 2 or more, preferably from 2 to 10, more preferably from 2 to 7, especially preferably from 2 to 4.

In addition, as the structure of the hydroxyl group-containing compound, it is particularly preferable that the structure in which each of two or more adjacent carbon atoms has a hydroxyl group is present in the terminal portion of a molecule.

Examples thereof include a compound represented by the formula (I):



wherein R^1 is a hydrocarbon group having 1 to 24 carbon atoms; X is a group represented by $(CH_2)_m$, wherein m is 0 or 1, oxygen atom, sulfur atom, COO group, OCO group, a group represented by NR^2 or $O(R^2O)P(O)O$, wherein R^2 is hydrogen atom or a hydrocarbon group having 1 to 24 carbon atoms; q is 0 or 1; and n is an integer of 1 to 4.

In the formula (I), the hydrocarbon group of R^1 may be either aliphatic or aromatic group, and the aliphatic group is preferable. The structure of the aliphatic group may be saturated or unsaturated, or linear or branched. From the viewpoint of suppressing dishing, a saturated structure is preferable, and a linear structure is preferable. In addition, the number of carbon atoms of the above hydrocarbon groups is preferably 1 or more, from the viewpoint of suppressing the dishing, and the number of

carbon atoms is preferably 24 or less, from the viewpoint of the solubility of the compound represented by the formula (I) in water. The number of carbon atoms is more preferably from 1 to 18, still more preferably from 2 to 12. Each of m and q is preferably 1. The number of carbon atoms of R² is preferably 12 or less, more preferably 8 or less, still more preferably 4 or less, from the viewpoint of suppressing the dishing. Especially, R² is preferably hydrogen atom or methyl group. n is preferably 2 or less, more preferably 1, from the viewpoint of suppressing the dishing.

In addition, the hydroxyl group-containing compound may have various functional groups other than hydroxyl group in a molecule. From the viewpoints of maintaining the polishing speed and suppressing the dishing, those compounds which do not contain carboxyl group, sulfonate group, primary amino group or phenolic hydroxyl group are preferable.

The hydroxyl group-containing compound has a molecular weight of preferably 5000 or less, more preferably 1000 or less, especially preferably 500 or less, from the viewpoints of maintaining the polishing speed and suppressing the dishing.

The hydroxyl group-containing compound has an acid dissociation constant pK_a in an aqueous solution of preferably 8 or more, more preferably 9 or more, especially preferably 10 or more, from the viewpoints of maintaining the polishing speed and suppressing the dishing. However, in a case where the hydroxyl group-containing compound has two or more dissociable functional groups in a molecule, pK_a referred herein is a first dissociation constant. In addition, the solubility of the hydroxyl group-containing compound at a pH usable for a polishing liquid composition is

such that the compound dissolves preferably at 0.5% by weight or more, more preferably at 1.0% by weight or more, in water at 25°C, from the viewpoint of formulating the hydroxyl group containing compound in an aqueous medium.

Concrete examples of these hydroxyl group-containing compounds include alkanediols such as 1,2-butanediol, 1,2-heptanediol, 1,2-hexanediol ("a" in Table 1 mentioned below), and 1,2-octanediol; alkanetriols such as 1,2,3-hexanetriol, 1,2,6-hexanetriol, and 1,2,3-heptanetriol; glyceryl ethers such as butyl glyceryl ether ("b" in Table 1 mentioned below), pentyl glyceryl ether, hexyl glyceryl ether, and octyl glyceryl ether; monoglycerides such as glyceryl monobutanoate, glyceryl monopentanoate, glyceryl monohexanoate, glyceryl monoheptanoate ("c" in Table 1 mentioned below), and glyceryl monooctanoate; partially esterified products prepared by carrying out esterification reaction of gluconic acid and an alcohol such as hexyl alcohol; compounds prepared by reacting glycidol with a monoalkylamine such as hexylamine or a dialkylamine such as dipropylamine ("d" in Table 1 mentioned below); diesters of tartaric acid such as diethyl tartrate, dibutyl tartrate, dipropyl tartrate ("e" in Table 1 mentioned below), and dihexyl tartrate; 1,2-cyclohexanediol, and the like. Among them, from the viewpoints of maintaining the polishing speed and suppressing the dishing, the alkanediols and the glyceryl ethers are preferable. These hydroxyl group-containing compounds may be used alone or in admixture of two or more kinds.

The amount of the hydroxyl group-containing compounds formulated is preferably from 0.01 to 30% by weight, more preferably from 0.05 to 5%

by weight, still more preferably from 0.1 to 3% by weight, of the polishing liquid composition 1-1, from the viewpoints of maintaining the polishing speed and suppressing the dishing.

Water usable in this embodiment is used as a medium. Its amount formulated is preferably from 60 to 99.99% by weight, more preferably from 70 to 99.4% by weight, still more preferably from 80 to 99.0% by weight, of the polishing liquid composition 1-1, from the viewpoint of efficiently polishing the substrate to be polished.

The polishing liquid composition 1-1 of this embodiment having the composition described above has a pH of preferably from 2 to 11, more preferably from 2 to 7, still more preferably from 2 to 6, especially preferably 3 to 5, from the viewpoints of keeping a polishing speed at a practical level, suppressing the dishing, and removing fine scratch damages on the surface. In order to adjust the pH within the above ranges, an inorganic acid such as nitric acid or sulfuric acid, an organic acid, a basic substance such as potassium hydroxide, sodium hydroxide, ammonia, or an organic amine can be appropriately added, as occasion demands.

In addition, the polishing liquid composition 1-1 of this embodiment may further comprise an organic acid.

In this embodiment, since the organic acid is used, the organic acid forms a complex with or binds to various metals, especially copper, constituting the metal layer, so that the metal layer is made brittle, whereby exhibiting an effect that the removal of the metal layer is made easy during polishing.

In addition, especially, the organic acid can be used in combination

with a compound having a structure in which each of two or more adjacent carbon atoms has a hydroxyl group in a molecule, whereby maintaining the polishing speed and preventing the dishing.

The organic acid is an organic compound showing acidic property.

5 These organic compounds showing acidic property include those having functional groups such as carboxyl group, phosphonic group, phosphinic group, sulfonic group, sulfinic group, phenol group, enol group, thiophenol group, imido group, oxime group, aromatic sulfamide groups, and primary and secondary nitro groups.

10 The organic acid usable in this embodiment has a molecular weight of preferably 1000 or less, more preferably 500 or less.

The organic acid having carboxyl group includes monocarboxylic acids having 1 to 24 carbon atoms, dicarboxylic acids, hydroxycarboxylic acids and aminocarboxylic acids are preferable, from the viewpoint of the solubility in water. The number of carbon atoms is more preferably 1 to 18 carbon atoms, still more preferably 1 to 12 carbon atoms, especially preferably 1 to 8 carbon atoms, most preferably 1 to 6 carbon atoms.

Concrete examples thereof include monocarboxylic acids, such as formic acid, acetic acid, propionic acid, butyric acid, valerianic acid, caproic acid, and pyruvic acid; dicarboxylic acids such as oxalic acid, malonic acid, succinic acid, glutaric acid, and adipic acid; hydroxycarboxylic acids such as gluconic acid, tartaric acid, glycolic acid, lactic acid, citric acid, and malic acid; aminocarboxylic acid such as nitrilotriacetic acid. The organic acid having phosphonic group includes aminotri(methylenephosphonic acid),

25 1-hydroxyethylidene-1,1-diphosphonic acid,

ethylenediaminetetra(methylenephosphonic acid),
hexamethylenediaminetetra(methylenephosphonic acid),
diethylenetriaminepenta(methylenephosphonic acid); the organic acid
having phosphinic group includes ethyl phosphite, and the like; the organic
5 acid having sulfonic group includes methanesulfonic acid, benzenesulfonic
acid, *p*-toluenesulfonic acid, naphthalenesulfonic acid, and the like; the
organic acid having sulfinic group includes benzenesulfinic acid,
p-toluenesulfinic acid, and the like. Among them, the organic acids having
carboxyl group are preferable. More concretely, monocarboxylic acids,
10 dicarboxylic acids, hydroxycarboxylic acids and aminocarboxylic acids are
preferable, and acetic acid, oxalic acid, succinic acid, glycolic acid, lactic acid,
citric acid, malic acid, tartaric acid, gluconic acid and nitrilotriacetic acid
are more preferable, and glycolic acid and gluconic acid are still more
preferable. These organic acids may be used alone or in admixture of two or
15 more kinds.

The organic acid is used in a state in which water is used as a
medium in the polishing liquid composition 1-1. The amount of the organic
acid formulated in the polishing liquid composition 1-1 can be widely
selected for the purposes of securing the polishing speed at a practical level
20 to remove the metal layer, and preventing excessive etching of the metal
layer. The amount of the organic acid formulated is, for instance, preferably
from 0.1 to 10% by weight, more preferably from 0.2 to 8% by weight, still
more preferably from 0.3 to 5% by weight.

In addition, among the above organic acids, a compound capable of
25 dissolving and etching a metal, especially copper in the copresence of an

aqueous medium, and having an etching speed "a" of 3 Å/min or more, as obtained by the following etching test A can be used as an etching agent. Specifically, in the etching test A, first a copper ribbon (commercially available from K.K. Nirako; thickness: 0.10 mm, width: 6 mm) having a length of 100 mm is furnished, and the surface dirt or the like is wiped of with a sheet of paper. Thereafter, the copper ribbon is subjected to ultrasonic cleaning for one minute in a state of being immersed in normal hexane, and thereafter the surface is sufficiently degreased and dried. Subsequently, its metal test piece is coiled into a helical form so that an entire surface of the ribbon is immersed in the polishing liquid, to give a metal test piece before test. The weight before immersion is determined by accurate balance.

Next, the etching agent is diluted to give a 2% by weight aqueous solution thereof. Further, 100 g of the etching solution of which pH is adjusted to 8 ± 0.5 with aqueous ammonia is furnished in a 150 cc beaker (K.K. Teraoka, 150 cc disposable cup), and the above metal test piece is immersed at 25°C for 12 hours. During immersion, the etching solution is stirred with a magnetic stirrer, to an extent that the copper ribbon rotates along with the flow of the etching solution. After the test, the copper ribbon surface is sufficiently wiped off, and its weight is again determined by accurate balance, to give a weight after the test. The amount of reduced thickness of copper is calculated from the weight loss of the copper ribbon before and after the test, and the resulting amount is divided by the etching time period to determine an etching speed "a." From the viewpoint of obtaining a practical polishing speed, an etching agent having an etching

speed "a" obtainable from the above etching test of 3 Å/min or more is preferable, more preferably 5 Å/min or more, still more preferably 10 Å/min or more. The etching speed "a" in this case may be an etching speed of two or more etching agents used in combination.

5 In addition, as the etching agent, an inorganic acid which is capable of dissolving and etching a metal, especially copper, in the copresence of an aqueous medium, and having an etching speed "a" obtainable from the above etching test A of 3 Å/min or more can be used.

10 Among the organic acids, from the viewpoint of having an appropriate etching speed, preferable etching agents include one or more compounds selected from the group consisting of the following A to D. Also, the following inorganic acids E are usable as an etching agent.

A: aliphatic organic acids having 6 or less carbon atoms and one to three carboxyl groups;

15 B: aromatic organic acids having 7 to 10 carbon atoms and one to four carboxyl groups;

C: organic acids having 6 or less carbon atoms and one to four phosphonic groups;

20 D: polyaminocarboxylic acids having in a molecule two or more structures represented by the formula (III):



and

E: inorganic acids.

Concretely, the aliphatic organic acid of the group A having 6 or less carbon atoms and one to three carboxyl groups includes monocarboxylic acids, such as formic acid, acetic acid, and propionic acid; polycarboxylic acids, such as oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, and tricarballic acid; hydroxycarboxylic acids such as glycolic acid, lactic acid, 2-hydroxypropionic acid, malic acid, tartaric acid, citric acid, and gluconic acid; amino acids such as glycine, alanine, and aspartic acid; and the like. The aromatic organic acid of the group B having 7 to 10 carbon atoms and one to four carboxyl groups includes benzoic acid, phthalic acid, trimellitic acid, pyromellitic acid, mandelic acid, salicylic acid, and the like. The organic acid of the group C having 6 or less carbon atoms and one to four phosphonic groups includes phosphonic acids such as methylphosphonic acid and phenylphosphonic acid; phosphinic acids such as methylphosphinic acid and phenylphosphinic acid; phosphonic acid esters such as methyl ester of phosphonic acid; aminophosphonic acids such as aminotri(methylenephosphonic acid) and 1-hydroxyethylidene-1-diphosphonic acid; and the like. The polyaminocarboxylic acid of the group D having in a molecule two or more structures represented by the formula (III) includes ethylenediaminetetraacetic acid, nitrilotriacetic acid, diethylenediaminepentaacetic acid, triethylenetetraminehexaacetic acid, hydroxyethylethylenediaminetetraacetic acid, and the like. The inorganic acid of the group E includes hydrochloric acid, perchloric acid, sulfuric acid, nitric acid, phosphoric acid, phosphonic acid, phosphinic acid, and the like. Among them, from the viewpoint of the polishing speed, preferable are the

polycarboxylic acids or hydroxycarboxylic acids belonging to the group A or B; aminophosphonic acids belonging to the group C; polyaminocarboxylic acids of the group D having in a molecule two or more structures represented by the formula (III); and hydrochloric acid, sulfuric acid, nitric acid, and phosphoric acid belonging to the group E. More preferable are oxalic acid, succinic acid, glycolic acid, lactic acid, citric acid, malic acid, gluconic acid, phthalic acid, aminotri(methylenephosphonic acid), 1-hydroxyethylidene-1-diphosphonic acid, ethylenediaminetetraacetic acid, hydrochloric acid, and sulfuric acid. These etching agents may be used alone or in admixture of two or more kinds. In this embodiment, glycolic acid and gluconic acid are especially preferable.

These etching agents, for instance, have the following etching speed "a": Glycolic acid, 60 Å/min; citric acid, 25 Å/min; phthalic acid, 50 Å/min; aminotri(methylenephosphonic acid), 10 Å/min; ethylenediaminetetraacetic acid, 30 Å/min; acetic acid, 70 Å/min, glycine, 40 Å/min; hydrochloric acid, 400 Å/min; and sulfuric acid, 100 Å/min.

When the etching agent usable in this embodiment is used in the preparation of a polishing liquid composition, with proviso that the composition does not comprise the hydroxyl group-containing compound, in which an oxidizing agent, abrasive grains, and the like are further copresent, it is preferable to adjust the kinds, contents, and the like, so that the etching liquid composition has an etching speed "b" obtainable from the following etching test B of 20 Å/min or more. The etching test B is carried out in the same procedures as in the etching test A, except that the copper ribbon is immersed in a polishing liquid composition comprising, as an

etching solution of the etching test A, water, an abrasive and an etching agent, and, if necessary, an oxidizing agent, at room temperature (25°C) for 2 hours, and that the pH is adjusted to 4.0 ± 0.5 . The etching speed obtained by the etching test B is referred to as "etching speed 'b'." From the viewpoint of obtaining a practical polishing speed, the etching speed "b" obtainable from the above etching test B is preferably 20 Å/min or more, more preferably 30 Å/min or more, still more preferably 50 Å/min or more. The etching speed "b" in this case may be an etching speed of a polishing liquid composition in which two or more etching agents are used in combination.

In this embodiment, since the etching agent is used, the etching agent forms a complex with or binds to various metals, especially copper, constituting the metal layer, so that the removal of the metals is made easy as water-soluble salts and/or chelated compounds, whereby exhibiting an effect that the polishing speed of the metal layer is increased during polishing.

The amount of the etching agent formulated in the polishing liquid composition 1-1 can be variously selected in order to secure the polishing speed at a practical level to remove the metal layer, and to prevent excessive etching of the metal layer. The amount of the etching agent formulated is preferably from 0.1 to 10% by weight, more preferably from 0.2 to 8% by weight, still more preferably from 0.3 to 5% by weight, of the polishing liquid composition 1-1.

Embodiment 2

In this embodiment, one of the largest features resides in that the above aliphatic carboxylic acid having 7 to 24 carbon atoms and/or a salt thereof and the etching agent are used in combination. Since the aliphatic carboxylic acid having 7 to 24 carbon atoms and/or a salt thereof has an action of lowering the etching speed, the polishing speed at a practical level can be maintained and excessive etching of the metal film in the metal layer can be prevented by the use of a polishing liquid composition comprising these compounds and the etching agent. Therefore, there is exhibited an excellent effect that the polishing surface without defects such as dishing can be obtained.

From the viewpoints of maintaining the polishing speed and suppressing the dishing, the aliphatic carboxylic acid and/or a salt thereof has 7 to 24 carbon atoms. Further, from the viewpoints of keeping the solubility in the polishing liquid, the low-foamability, and the polishing speed at practical levels, and suppressing the dishing, these compounds have preferably from 7 to 20 carbon atoms, more preferably from 7 to 16 carbon atoms, still more preferably from 7 to 12 carbon atoms, especially preferably from 7 to 10 carbon atoms.

In addition, the hydrocarbon group of the aliphatic carboxylic acid and/or a salt thereof may be saturated or unsaturated, or linear or branched.

In addition, the salts of the aliphatic carboxylic acids may be any of ammonium salts, salts of organic amines, and alkali metal salts. From the viewpoint of preventing staining of the semiconductors, ammonium salts, and salts of organic amines such as salts of monoethanolamine, salts of diethanolamine, salts of triethanolamine, and salts of triethylamine.

Concrete examples of these aliphatic carboxylic acids and salts thereof include linear, saturated aliphatic carboxylic acids such as heptanoic acid, octanoic acid, nonanoic acid, decanoic acid, lauric acid, myristic acid, palmitic acid, stearic acid, and eicosanoic acid; linear, unsaturated aliphatic carboxylic acids such as heptenoic acid, octenoic acid, decenoic acid, dodecenoic, and oleic acid; branched, saturated aliphatic carboxylic acids such as 2-methylhexanoic acid, 2-ethylhexanoic acid, 3,5-dimethylhexanoic acid, 3,5,5-trimethylhexanoic acid, and isodecanoic acid; and ammonium salts, salts of organic amines, alkali metal salts of these aliphatic carboxylic acids, and the like. Among them, from the viewpoints of the polishing speed and the suppression of the dishing, the linear or branched, saturated aliphatic carboxylic acids and ammonium salts thereof are preferable, and from the viewpoint of the solubility in the polishing liquid and the low foamability, heptanoic acid or ammonium salt thereof, octanoic acid or ammonium salt thereof, nonanoic acid or ammonium salt thereof, and decanoic acid or ammonium salt thereof are especially preferable. These aliphatic carboxylic acids having 7 to 24 carbon atoms and salts thereof may be used alone or in admixture of two or more kinds.

The amount of the aliphatic carboxylic acid and a salt thereof formulated is preferably from 0.01 to 30% by weight, more preferably from 0.02 to 10% by weight, still more preferably from 0.03 to 5% by weight, of the polishing liquid composition 1-2, from the viewpoint of maintaining the polishing speed, and suppressing the dishing.

The etching agent usable in this embodiment is capable of dissolving and etching a metal, especially copper, in the copresence of an aqueous

medium, and is a compound having an etching speed "a" of 3 Å/min or more, as obtained by the etching test A described in Embodiment 1. From the viewpoint of obtaining a practical polishing speed, an etching agent having an etching speed "a" obtainable from the above etching test A of 3 Å/min or more is preferable, more preferably 5 Å/min or more, still more preferably 10 Å/min or more. The etching speed "a" in this case may be an etching speed of two or more etching agents used in combination.

Preferable etching agents include the same ones as those in Embodiment 1 mentioned above. These etching agents may be used alone or in admixture of two or more kinds. In this embodiment, glycolic acid, citric acid, and aminotri(methylenephosphonic acid) are especially preferable. Incidentally, the etching speed "a" of the aliphatic carboxylic acid having 7 to 24 carbon atoms is 2 Å/min or less, and as compared to the etching agent, the etching strength is none or almost not found.

When the etching agent usable in this embodiment is used in the preparation of a polishing liquid composition, with proviso that the composition does not comprise the aliphatic carboxylic acid having 7 to 24 carbon atoms and/or a salt thereof, in which an oxidizing agent, abrasive grains, and the like are further copresent, it is preferable to adjust the kinds, contents, and the like, so that the polishing liquid composition has an etching speed "c" obtainable from the following etching test C of 20 Å/min or more. The etching test C is carried out in the same procedures as in the etching test A, except that the copper ribbon is immersed in a polishing liquid composition comprising, as an etching solution of the etching test A, water, an abrasive and an etching agent, and, if necessary, an oxidizing

agent, at room temperature (25°C) for 2 hours. The etching speed obtained by the etching test C is referred to as "etching speed 'c'." From the viewpoint of obtaining a practical polishing speed, the etching speed "c" obtainable from the above etching test C is preferably 20 Å/min or more, more preferably 30 Å/min or more, still more preferably 50 Å/min or more. The etching speed "c" in this case may be an etching speed of a polishing liquid composition in which two or more etching agents are used in combination.

In this embodiment, since the etching agent is used, the etching agent forms a complex with or binds to various metals, especially copper, constituting the metal layer, so that the removal of the metals is made easy as water-soluble salts and/or chelated compounds, whereby exhibiting an effect that the polishing speed of the metal layer is increased during polishing.

The amount of the etching agent formulated in the polishing liquid composition 1-2 can be variously selected in order to secure the polishing speed at a practical level to remove the metal layer, and to prevent excessive etching of the metal layer. The amount of the etching agent formulated is preferably from 0.1 to 10% by weight, more preferably from 0.2 to 8% by weight, still more preferably from 0.3 to 5% by weight, of the polishing liquid composition 1-2.

Water usable in this embodiment is used as a medium. The amount of water formulated is preferably from 60 to 99.89% by weight, more preferably from 70 to 99.4% by weight, still more preferably from 80 to 99% by weight, of the polishing liquid composition 1-2, from the viewpoint of

efficiently polishing the substrate to be polished.

The polishing liquid composition 1-2 of this embodiment having the composition described above has a pH of preferably 10 or less, more preferably from 2 to 9.5, still more preferably from 4 to 9, especially preferably from 7 to 9, from the viewpoints of keeping a polishing speed at a practical level, suppressing the dishing, and removing fine scratch damages on the surface. In order to adjust the pH within the above ranges, an inorganic acid such as nitric acid or sulfuric acid, an organic acid, a basic substance such as potassium hydroxide, sodium hydroxide, ammonia, or an organic amine can be appropriately added, as occasion demands.

Embodiment 3

In this embodiment, one of the largest features resides in that the above amine compound represented by the general formula (II) and/or a salt thereof and the etching agent are used in combination. The polishing speed at a practical level can be maintained and excessive etching of the metal film of the metal layer can be prevented by the use of a polishing liquid composition comprising the amine compound and/or a salt thereof, and the etching agent. Therefore, there is exhibited an excellent effect that the polishing surface without defects such as dishing can be obtained.

The above amine compound represented by the general formula (II) and/or a salt thereof usable in this embodiment is preferable, from the viewpoints of maintaining the polishing speed and suppressing the dishing. Further, from the viewpoints of keeping the low foamability and the polishing speed at practical levels and suppressing the dishing, in the

formula, R^3 is preferably a linear or branched, alkyl or alkenyl group having 5 to 14 carbon atoms, more preferably a linear or branched, alkyl or alkenyl group having 6 to 12 carbon atoms, still more preferably a linear or branched, alkyl or alkenyl group having 7 to 10 carbon atoms. Each of R^4 and R^5 is preferably hydrogen, a linear alkyl group having 1 or 2 carbon atoms, a group represented by $H-(OR^6)_Z$, wherein R^6 is an alkylene group having 2 carbon atoms and Z is a number from 1 to 4, and more preferably hydrogen atom, methyl group and hydroxyethyl group.

In addition, the salt of the amine compound may be either a salt with an inorganic acid or a salt with an organic acid, and those which are a salt with an inorganic acid or organic acid which is usable as an etching agent are preferable. Further, from the viewpoint of preventing staining of the semiconductor, salts of organic acids are more preferable among the etching agents.

Concrete examples of these amine compounds and salts thereof include linear monoalkylamines such as butylamine, pentylamine, hexylamine, heptylamine, octylamine, nonylamine, decylamine, laurylamine, myristylamine, and stearylamine; linear monoalkenylamines such as oleylamine; branched monoalkylamines such as 2-ethylhexylamine; dialkylamines such as dihexylamine and dioctylamine; trialkylamines such as dimethyloctylamine, dimethyldecylamine, and dimethyldodecylamine; alkylalkanolamines such as octyldiethanolamine, decyldiethanolamine, and dodecyldiethanolamine; and carboxylates, phosphates, hydrochlorides, sulfates, nitrates, and the like of these amine compounds. Among them, from the viewpoints of maintaining the polishing speed and suppressing the

dishing, monoalkylamines, monoalkyldimethylamines, and monoalkyldiethanolamines, each of which is linear or branched, and carboxylates thereof are preferable. Further, from the viewpoint of the low foamability, more preferable are heptylamine, octylamine, nonylamine, dimethyloctylamine, dimethyldecylamine, dimethyldodecylamine octyldiethanolamine, decyldiethanolamine, and carboxylates thereof. These amine compounds and salts thereof may be used alone or in admixture of two or more kinds.

The amount of the amine compound and a salt thereof formulated is preferably from 0.01 to 30% by weight, more preferably from 0.02 to 10% by weight, still more preferably from 0.03 to 5% by weight, of the polishing liquid composition 1-3, from the viewpoint of maintaining the polishing speed, and suppressing the dishing.

The etching agent usable in this embodiment is capable of dissolving and etching a metal, especially copper, in the copresence of an aqueous medium, and is a compound having an etching speed "a" of 3 Å/min or more, as obtained by the etching test A described in Embodiment 1. From the viewpoint of obtaining a practical polishing speed, an etching agent having an etching speed "a" obtainable from the above etching test A of 3 Å/min or more is preferable, more preferably 5 Å/min or more, still more preferably 10 Å/min or more. The etching speed "a" in this case may be an etching speed of two or more etching agents used in combination.

Preferable etching agents include the same ones as those in Embodiment 1 mentioned above. These etching agents may be used alone or in admixture of two or more kinds. In this embodiment, glycolic acid,

citric acid, and aminotri(methylenephosphonic acid) are especially preferable.

When the etching agent usable in this embodiment is used in the preparation of a polishing liquid composition, with proviso that the composition does not comprise the above amine compound or a salt thereof, in which an oxidizing agent, abrasive grains, and the like are further copresent, it is preferable to adjust the kinds, contents, and the like, so that the polishing liquid composition has an etching speed "c" obtainable from the etching test C described in Embodiment 2 of 20 Å/min or more. From the viewpoint of obtaining a practical polishing speed, the etching speed "c" obtainable from the above etching test C is preferably 20 Å/min or more, more preferably 30 Å/min or more, still more preferably 50 Å/min or more. The etching speed "c" in this case may be an etching speed of a polishing liquid composition in which two or more etching agents are used in combination.

In this embodiment, since the etching agent is used, the etching agent forms a complex with or binds to various metals, especially copper, constituting the metal layer, so that the removal of the metals is made easy as water-soluble salts and/or chelated compounds, whereby exhibiting an effect that the polishing speed of the metal layer is increased during polishing.

The amount of the etching agent formulated in the polishing liquid composition 1-3 can be variously selected in order to secure the polishing speed at a practical level to remove the metal layer, and to prevent excessive etching of the metal layer. The amount of the etching agent

formulated is preferably from 0.1 to 10% by weight, more preferably from 0.2 to 8% by weight, still more preferably from 0.3 to 5% by weight, of the polishing liquid composition 1-3.

Water usable in this embodiment is used as a medium. The amount thereof is preferably from 60 to 99.89% by weight, more preferably from 70 to 99.4% by weight, still more preferably from 80 to 99% by weight, of the polishing liquid composition 1-3, from the viewpoint of efficiently polishing the substrate to be polished.

The polishing liquid composition 1-3 of this embodiment having the composition described above has a pH of preferably 10 or less, more preferably from 2 to 9.5, still more preferably from 4 to 9, especially preferably from 5 to 9, from the viewpoints of keeping a polishing speed at a practical level, suppressing the dishing, and removing fine scratch damages on the surface. In order to adjust the pH within the above ranges, an inorganic acid such as nitric acid or sulfuric acid, an organic acid, a basic substance such as potassium hydroxide, sodium hydroxide, ammonia, or an organic amine can be appropriately added, as occasion demands.

The polishing liquid composition 2 comprises one of the polishing liquid compositions 1-1 to 1-3 (hereinafter collectively referred to as "the polishing liquid composition 1"), and further comprising an oxidizing agent. The oxidizing agent usable in the present invention refers to those oxidizing a metal. In the present invention, it is thought that the metal layer is oxidized by the use of the oxidizing agent, whereby an effect of accelerating the mechanical polishing effect of the metal layer is exhibited.

The oxidizing agent includes, peroxides; permanganic acid or salts

thereof; chromic acid or salts thereof; nitric acid or salts thereof; peroxo acid or salts thereof; oxyacid or salts thereof; metal salts; sulfuric acid, and the like.

As concrete examples thereof, the peroxide includes hydrogen
5 peroxide, sodium peroxide, barium peroxide, and the like; the permanganic acid or salts thereof include potassium permanganate, and the like; chromic acid or salts thereof include metal salts of chromic acid, metal salts of dichromic acid, and the like; the nitrates include iron (III) nitrate, ammonium nitrate, and the like; the peroxo acid or salts thereof include
10 peroxodisulfuric acid, ammonium peroxodisulfate, metal salts of peroxodisulfuric acid, peroxophosphoric acid, peroxosulfuric acid, sodium peroxoborate, performic acid, peracetic acid, perbenzoic acid, perphthalic acid, and the like; the oxyacid or salts thereof include hypochlorous acid, hypobromous acid, hypoiodous acid, chloric acid, bromic acid, iodic acid,
15 perchloric acid, sodium hypochlorite, calcium hypochlorite, and the like; metal salts include iron (III) chloride, iron (III) sulfate, iron (III) citrate, ammonium iron (III) sulfate, and the like. Among the oxidizing agents, hydrogen peroxide, iron (III) nitrate, peracetic acid, ammonium peroxodisulfate, iron (III) sulfate and ammonium iron (III) sulfate are
20 preferable, and especially hydrogen peroxide is preferable. These oxidizing agents may be used alone or in admixture of two or more kinds.

The oxidizing agent is used in a state in which water is used as a medium in the polishing liquid composition 2. The amount of the oxidizing agent formulated is preferably from 0.1 to 60% by weight, more preferably
25 from 0.2 to 50% by weight, still more preferably from 0.3 to 30% by weight,

especially preferably from 0.3 to 10% by weight, of the polishing liquid composition 2, from the viewpoint of obtaining the polishing speed at a practical level by rapid oxidation of the metal layer.

In addition, when the polishing liquid composition 2 is prepared from the polishing liquid composition 1-1, the amount of the hydroxyl group-containing compound formulated is preferably from 0.01 to 30% by weight, more preferably from 0.05 to 5% by weight, still more preferably from 0.1 to 3% by weight, of the polishing liquid composition 2. The amount of water formulated is preferably from 40 to 99.89% by weight, more preferably from 70 to 99.4% by weight, still more preferably from 80 to 99% by weight, of the polishing liquid composition 2. The pH of the polishing liquid composition 2 having the above composition, which is the same as the polishing liquid composition 1-1, is preferably from 2 to 11, more preferably from 2 to 7, still more preferably 2 to 6, especially preferably from 3 to 5, from the viewpoints of keeping the polishing speed at a practical level, suppressing the dishing, and removing the fine scratch damages on the surface. In order to adjust the pH to the above-specified range, there may be added at an appropriate timing an inorganic acid such as nitric acid or sulfuric acid, an organic acid, or a basic substance such as potassium hydroxide, sodium hydroxide, ammonia, or an organic amine.

In addition, when the polishing liquid composition 2 is prepared from the polishing liquid composition 1-2, the amount of the aliphatic carboxylic acid having 7 to 24 carbon atoms and/or salts thereof formulated is preferably from 0.01 to 30% by weight, more preferably from 0.02 to 10% by weight, still more preferably from 0.03 to 5% by weight, of the polishing

liquid composition 2. The amount of the etching agent formulated is preferably from 0.1 to 10% by weight, more preferably from 0.2 to 8% by weight, still more preferably from 0.3 to 5% by weight, of the polishing liquid composition 2. The amount of water formulated is preferably from 39.89 to 99.79% by weight, more preferably from 70 to 99.4% by weight, still more preferably from 80 to 99% by weight, of the polishing liquid composition 2. The pH of the polishing liquid composition 2 having the above composition, which is the same as the polishing liquid composition 1-2, is preferably 10 or less, more preferably from 2 to 9.5, still more preferably 4 to 9, especially preferably from 7 to 9, from the viewpoints of keeping the polishing speed at a practical level, suppressing the dishing, and removing the fine scratch damages on the surface.

In addition, when the polishing liquid composition 2 is prepared from the polishing liquid composition 1-3, the amount of the amine compound and/or salts thereof formulated is preferably from 0.01 to 30% by weight, more preferably from 0.02 to 10% by weight, still more preferably from 0.03 to 5% by weight, of the polishing liquid composition 2. The amount of the etching agent formulated is preferably from 0.1 to 10% by weight, more preferably from 0.2 to 8% by weight, still more preferably from 0.3 to 5% by weight, of the polishing liquid composition 2. The amount of water formulated is preferably from 39.89 to 99.79% by weight, more preferably from 70 to 99.4% by weight, still more preferably from 80 to 99% by weight, of the polishing liquid composition 2. The pH of the polishing liquid composition 2 having the above composition, which is the same as the polishing liquid composition 1-3, is preferably 10 or less, more preferably

from 2 to 9.5, still more preferably 4 to 9, especially preferably from 5 to 9, from the viewpoints of keeping the polishing speed at a practical level, suppressing the dishing, and removing the fine scratch damages on the surface.

5 The polishing liquid compositions 1 and 2 of the present invention are effective for polishing processes using a fixed grinding wheel, a polishing pad in which abrasive grains are fixed to the pad, and the like. For instance, by the use of the polishing liquid compositions 1 and 2 of the present invention during polishing in the polishing process using the fixed grinding
10 wheel, the polishing speed can be maintained, and the dishing of the metal layer can be suppressed.

 The polishing liquid composition 3 of the present invention is one further comprising an abrasive to a polishing liquid composition 1 or 2, which is usable for a polishing process by loose abrasives.

15 As the abrasive, abrasives generally employed for polishing can be used. The abrasive includes, for instance, metals, carbides of metals or metalloids, nitrides of metals or metalloids, oxides of metals or metalloids, borides of metals or metalloids, diamond, and the like. The metals or metalloids include those elements belonging to the Groups 3A, 4A, 5A, 3B,
20 4B, 5B, 6B, 7B or 8B of the Periodic Table. Examples thereof include silicon dioxide, aluminum oxide, cerium oxide, titanium oxide, zirconium oxide, silicon nitride, manganese dioxide, silicon carbide, zinc oxide, diamond, and magnesium oxide. Among them, silicon dioxide, aluminum oxide and cerium oxide are preferable. As concrete examples thereof, the silicon
25 dioxide includes colloidal silica particles, fumed silica particles, surface-

modified silica particles, and the like; the aluminum oxide includes α -alumina particles, γ -alumina particles, δ -alumina particles, θ -alumina particles, η -alumina particles, amorphous alumina particles, and other fumed alumina or colloidal alumina prepared by different process; the cerium oxide includes ones having oxidation state of 3 or 4, of which crystal system is hexagonal system, isometric system, or face-centered cubic system, and the like. The silicon dioxide is especially preferable. These abrasives may be used alone or in admixture of two or more kinds.

The abrasive has a primary average particle size of preferably from 5 to 1000 nm, more preferably from 10 to 500 nm, still more preferably from 20 to 300 nm, especially preferably from 50 to 200 nm, most preferably from 50 to 100 nm. The lower limit of the average particle size is preferably 5 nm or more, from the viewpoint of maintaining a given polishing speed, and the upper limit thereof is preferably 1000 nm or less, from the viewpoint of preventing the generation of scratches on the surface of the substrate to be polished.

Especially when the silicon dioxide is used as an abrasive, the silicon dioxide has a primary average particle size of 5 nm or more, preferably 10 nm or more, more preferably 20 nm or more, from the viewpoint of improving the polishing speed.

Incidentally, the primary average particle size of the abrasive is determined by adding 0.1 g of the abrasive to 100 g of a 0.1% aqueous solution of sodium polystyrenesulfonate, thereafter applying ultrasonic waves, to disperse the abrasive, and measuring image analysis of the dispersion by observing with a transmission electron microscope.

When the polishing liquid composition 3 is used when forming interconnection of a semiconductor device, especially preferably usable abrasives are silica particles having purity of preferably 98% by weight or more, more preferably 99% by weight or more, especially preferably 99.9% by weight or more. The abrasive includes fumed silica prepared by
5 subjecting a volatile silicon compound such as silicon tetrachloride to high-temperature hydrolysis in oxyhydrogen flame; or colloidal silica obtained by a process in which an alkali silicate or ethyl silicate is used as a starting material.

10 Incidentally, the purity of the above abrasive is obtained as follows. Specifically, the purity can be determined by dissolving 1 to 3 g of an abrasive in an acid or an aqueous alkali, and quantifying silicon ions by ICP (plasma emission analysis).

15 The abrasive is used in a so-called "slurry state" using water as a medium in the polishing liquid composition 3. The amount of the abrasive formulated in the polishing liquid composition 3 can be variously selected depending upon the viscosity of the polishing liquid composition and the required quality of the substrate to be polished, and the like. The amount of the abrasive formulated is preferably from 0.01 to 30% by weight, more
20 preferably from 0.02 to 20% by weight, still more preferably from 0.1 to 20% by weight, especially preferably from 1.0 to 10% by weight, of the polishing liquid composition 3.

25 In addition, when the polishing liquid composition 3 is prepared from the polishing liquid composition 1-1 (hereinafter referred to as "polishing liquid composition 3-1"), the amount of the hydroxyl-group containing

compound formulated is preferably from 0.01 to 30% by weight, more preferably from 0.05 to 5% by weight, still more preferably from 0.1 to 3% by weight, of the polishing liquid composition 3-1, from the viewpoints of maintaining the polishing speed and suppressing the dishing.

5 The amount of the organic acid formulated in the polishing liquid composition 3-1 can be variously selected in order to secure a polishing speed at a practical level for removal of the metal layer, and to prevent excessive etching of the metal layer. The amount is, for instance, preferably from 0.1 to 10% by weight, more preferably from 0.2 to 8% by weight, still
10 more preferably from 0.3 to 5% by weight, of the polishing liquid composition 3-1.

 The amount of the oxidizing agent formulated is preferably from 0.1 to 60% by weight, more preferably from 0.2 to 50% by weight, still more preferably from 0.3 to 30% by weight, of the polishing liquid composition
15 3-1, from the viewpoint of obtaining a polishing speed at a practical level by rapid oxidation of the metal layer.

 The amount of water formulated is preferably from 40 to 99.98% by weight, more preferably from 60 to 99.4% by weight, still more preferably from 75 to 99% by weight, of the polishing liquid composition 3-1. The pH
20 of the polishing liquid composition 3-1 having the above composition, which is the same as the polishing liquid composition 1-1, is preferably from 2 to 11, more preferably from 2 to 7, still more preferably 2 to 6, especially preferably from 3 to 5, from the viewpoints of keeping the polishing speed at a practical level, suppressing the dishing, and removing the fine scratch
25 damages on the surface.

In addition, when the polishing liquid composition 3 is prepared from the polishing liquid composition 1-2 (hereinafter referred to as "polishing liquid composition 3-2"), the amount of the aliphatic carboxylic acid having 7 to 24 carbon atoms and/or salts thereof formulated is preferably from 0.01 to 30% by weight, more preferably from 0.02 to 10% by weight, still more preferably from 0.03 to 5% by weight, of the polishing liquid composition 3-2, from the viewpoints of maintaining the polishing speed and suppressing the dishing.

The amount of the etching agent formulated in the polishing liquid composition 3-2 can be variously selected in order to secure a polishing speed at a practical level for removal of the metal layer, and to prevent excessive etching of the metal layer. The amount formulated is, for instance, preferably from 0.1 to 10% by weight, more preferably from 0.2 to 8% by weight, still more preferably from 0.3 to 5% by weight, of the polishing liquid composition 3-2.

The amount of the oxidizing agent formulated is preferably from 0.1 to 60% by weight, more preferably from 0.2 to 50% by weight, still more preferably from 0.3 to 30% by weight, especially preferably from 0.3 to 10% by weight, of the polishing liquid composition 3-2, from the viewpoint of obtaining a polishing speed at a practical level by rapid oxidation of the metal layer.

The amount of water formulated is preferably from 39.88 to 99.88% by weight, more preferably from 60 to 99.4% by weight, still more preferably from 75 to 99% by weight, of the polishing liquid composition 3-2. The pH of the polishing liquid composition 3-2 having the above composition, which

is the same as the polishing liquid composition 1-2, is preferably 10 or less, more preferably from 2 to 9.5, still more preferably from 4 to 9, especially preferably from 7 to 9, from the viewpoints of keeping the polishing speed at a practical level, suppressing the dishing, and removing the fine scratch damages on the surface. In order to adjust the pH within the above ranges, an inorganic acid such as nitric acid or sulfuric acid, an organic acid, a basic substance such as potassium hydroxide, sodium hydroxide, ammonia, or an organic amine can be appropriately added, as occasion demands.

In addition, when the polishing liquid composition 3 is prepared from the polishing liquid composition 1-3 (hereinafter referred to as "polishing liquid composition 3-3"), the amount of the amine compound and/or salts thereof formulated is preferably from 0.01 to 30% by weight, more preferably from 0.02 to 10% by weight, still more preferably from 0.03 to 5% by weight, of the polishing liquid composition 3-3, from the viewpoints of maintaining the polishing speed and suppressing the dishing.

The amount of the etching agent formulated in the polishing liquid composition 3-3 can be variously selected in order to secure a polishing speed at a practical level for removal of the metal layer, and to prevent excessive etching of the metal layer. The amount formulated is, for instance, preferably from 0.1 to 10% by weight, more preferably from 0.2 to 8% by weight, still more preferably from 0.3 to 5% by weight, of the polishing liquid composition 3-3.

The amount of the oxidizing agent formulated is preferably from 0.1 to 60% by weight, more preferably from 0.2 to 50% by weight, still more preferably from 0.3 to 30% by weight, especially preferably from 0.3 to 10%

by weight, of the polishing liquid composition 3-3, from the viewpoint of obtaining a polishing speed at a practical level by rapid oxidation of the metal layer.

The amount of water formulated is preferably from 39.88 to 99.88% by weight, more preferably from 60 to 99.4% by weight, still more preferably from 75 to 99% by weight, of the polishing liquid composition 3-3. The pH of the polishing liquid composition 3-3 having the above composition, which is the same as the polishing liquid composition 1-3, is preferably 10 or less, more preferably from 2 to 9.5, still more preferably from 4 to 9, especially preferably from 5 to 9, from the viewpoints of keeping the polishing speed at a practical level, suppressing the dishing, and removing the fine scratch damages on the surface.

The polishing liquid compositions 1 to 3 of the present invention can be prepared in any manner without particular limitations, and for instance, a polishing liquid composition can be obtained by appropriately mixing each of the above components, and adjusting a pH. Concrete examples thereof are as follows.

The polishing liquid composition 1 of the present invention can be, for instance, prepared by the following procedures. First, an organic acid and/or an etching agent is added to a given amount of water, and a pH is adjusted to a given value. To the pH-adjusted aqueous solution of the organic acid and/or etching agent is added a given amount of an aqueous solution of a hydroxyl group-containing compound; or an aqueous solution of an aliphatic carboxylic acid having 7 to 24 carbon atoms and/or salts thereof, or an amine compound and/or salts thereof, the aqueous solution of

which pH is adjusted to a given level. A pH is finally adjusted, whereby a polishing liquid composition 1 can be obtained.

The polishing liquid composition 2 can be, for instance, prepared by the following procedures. First, an organic acid and/or an etching agent is added to a given amount of water, and a pH is adjusted to a given value. To the pH-adjusted aqueous solution of the organic acid and/or etching agent is added a given amount of an aqueous solution of a hydroxyl group-containing compound; or an aqueous solution of an aliphatic carboxylic acid having 7 to 24 carbon atoms and/or salts thereof, or an amine compound and/or salts thereof, the aqueous solution of which pH is adjusted to a given level. An oxidizing agent is added before polishing, and a pH is finally adjusted, whereby a polishing liquid composition 2 can be obtained.

The polishing liquid composition 3 can be, for instance, prepared by the following procedures. First, an organic acid and/or an etching agent is added to a given amount of water, and a pH is adjusted to a given value. To the pH-adjusted aqueous solution of the organic acid and/or etching agent is added a given amount of an abrasive, and the mixture is sufficiently stirred so as to uniformly disperse the abrasives. Further, to the dispersion is added a given amount of an aqueous solution of a hydroxyl group-containing compound; or an aqueous solution of an aliphatic carboxylic acid having 7 to 24 carbon atoms and/or salts thereof, or an amine compound and/or salts thereof, the aqueous solution of which pH is adjusted to a given level. A given amount of an oxidizing agent is added before polishing as occasion demands, and a pH is finally adjusted, whereby a polishing liquid composition 3 can be obtained.

In addition, various polishing aids other than those listed above such as surfactants and dispersion stabilizers may be added to each of the polishing liquid compositions 1 to 3.

5 The polishing liquid composition of the present invention can be suitably used for the metal CMP in which a surface comprising an insulating layer and a metal layer is subject to polishing. The metal constituting the metal layer includes copper or copper alloys, aluminum or aluminum alloys, tungsten, and the like. Among these metals, especially when used for a process for forming embedded metal interconnection on a semiconductor substrate, copper or copper alloys are preferable. When the
10 polishing liquid composition of the present invention is used in the formation of the metal interconnection layer made of copper or copper alloys, there are exhibited remarkable effects of maintaining the polishing speed and suppressing the dishing of the embedded metal interconnection layer.

15 In addition, the material for forming the insulating layer may be any of organic or inorganic materials, and includes inorganic materials such as silicon dioxide, fluorinated silicon dioxide, hydrogen-containing SOG (spin-on glass), nitrides, such as tantalum nitride and titanium nitride; and organic materials such as organic SOG, polyimides, fluorinated polyimides,
20 methyl polysiloxanes, aromatic polyethers, hydrogensilsesquioxane, and fluorocarbons.

The shape of these substrates to be polished is preferably those having a shape of forming recesses of an interconnection shape on the insulating film surface of the semiconductor substrate, and sedimenting
25 metals on the insulating film including the recesses. In addition, there may

be provided a barrier film made of tantalum, titanium, or a nitride thereof between the insulating film and the metal layer. Especially when the metal layer is made of copper or copper alloys, it is preferable to provide the barrier film, whereby the diffusion of the copper to the insulating layer can be prevented.

The polishing liquid compositions 1 and 2 of the present invention are effective for a polishing processes using a fixed grinding wheel, a polishing pad in which abrasive grains are fixed to the pad, and the like. The polishing liquid composition 3 is effective for a polishing process by loose abrasives using a usual polishing pad made of urethane, and it is also effective for a polishing processes using a fixed grinding wheel, a polishing pad in which abrasive grains are fixed to the pad, and the like.

The process for polishing a surface to be polished comprising an insulating layer and a metal layer of the present invention comprises polishing a semiconductor substrate using the polishing liquid composition of the present invention, thereby smoothening the polished surface.

In addition, the process for manufacturing a semiconductor substrate of the present invention comprises polishing a semiconductor surface comprising an insulating layer and a metal layer using the polishing liquid composition of the present invention, whereby the polishing speed of the metal layer can be maintained and the dishing of the embedded metal interconnection layer can be suppressed. Therefore, the process can be suitably applied to a process for manufacturing the semiconductor substrate.

Examples I-1 to I-14 and Comparative Examples I-1 to I-4

Compounds having a structure in which each of two or more adjacent carbon atoms has hydroxyl group in a molecule (hydroxyl group-containing compound) used in Examples I-1 to I-14 are shown in Table 1. The hydroxyl group-containing compound shown in Table 1, the organic acid shown in Table 2, and hydrogen peroxide were mixed so as to have the proportion shown in Table 2. Further, 5% by weight portion of an abrasive shown in Table 2 and balance water were mixed with stirring, and thereafter a pH of the liquid mixture was adjusted to 4.0, to give a polishing liquid composition. Incidentally, each of the used abrasives was fumed silica (primary particle size: 50 nm) and colloidal silica (primary particle size: 30 nm). In addition, each of the hydroxyl group-containing compounds "a" to "e" of Table 1 dissolves in water at 1.0% by weight or more. The substrate to be polished was polished under the following conditions by single-sided polishing machine. Hereinbelow, the terms inside quotation marks are trade names.

Setting Conditions of Single-Sided Processing Machine

Single-sided processing machine used: single-sided polishing machine (disc size: 30 cm), manufactured by Engis.

Processing pressure: 29.4×10^3 Pa

Polishing Pad: upper layer: "IC1000" (manufactured by Rodel Nitta K.K.); and lower layer: "SUBA400" (manufactured by Rodel Nitta K.K.).

Disc rotational speed: 60 rpm

Work rotational speed: 50 rpm (rotating disc and work in the same direction)

Feeding flow rate for a polishing liquid composition: 100 ml/min

Polishing time period: 10 minutes

In addition, the properties of the polishing liquid composition such as relative polishing speed, relative etching speed, and dishing of the surface to be polished were evaluated in accordance with the following methods. The results thereof are shown in Table 2.

Relative Polishing Speed

The relative polishing speed is a value obtained by dividing the polishing speed of a polishing liquid composition by a polishing speed of a comparative example where the kinds and the amounts of an abrasive, an oxidizing agent and an etching agent were the same as the polishing liquid composition. The polishing speed was obtained by polishing a rolled copper plate of a diameter of 50 mm and a plate thickness of 1 mm, determining a change in the thickness before and after polishing, and dividing the resultant value by a polishing time. Incidentally, the thickness of the copper plate was measured by using a high-precision digital analyzer "MINIAX" commercially available from K. K. Tokyo Seimitsu. Here, each of the relative polishing speeds for Examples I-1 to I-5 and I-9 to I-10 was calculated based on Comparative Example I-1; each of the relative polishing speeds for Examples I-6 to I-8 was calculated based on Comparative Example I-2; and each of the relative polishing speeds for Examples I-13 to I-14 was calculated based on Comparative Example I-3. Incidentally, apart from the above, the relative polishing speed for

Examples I-11 was calculated based on Comparative Example I-1; the relative polishing speed for Examples I-12 was calculated based on Comparative Example I-2; and the relative polishing speed for Comparative Example I-4 was calculated based on Comparative Example I-1.

5

Relative Etching Speed

The relative etching speed is a value obtained by dividing an etching speed of the polishing liquid composition comprising the above hydroxyl group-containing compound by an etching speed "b" of a polishing liquid composition not containing the above hydroxyl group-containing compound but having the same kinds and the amounts of an abrasive, an oxidizing agent and an etching agent as the polishing liquid composition. Here, each of the relative etching speeds for Examples I-1 to I-5 and I-9 to I-10 was calculated based on Comparative Example I-1; each of the relative etching speeds for Examples I-6 to I-8 was calculated based on Comparative Example I-2; and each of the relative etching speeds for Examples I-13 to I-14 was calculated based on Comparative Example I-3. Incidentally, apart from the above, the relative etching speed for Examples I-11 was calculated based on Comparative Example I-1; the relative etching speed for Examples I-12 was calculated based on Comparative Example I-2; and the relative etching speed for Comparative Example I-4 was calculated based on Comparative Example I-1. Incidentally, each the etching speeds for each the polishing liquid compositions of Examples I-1 to I-14 and Comparative Example I-4 was a value measured under the same conditions as the above etching test B described above, except for using these polishing

10

15

20

25

10030424-01002

liquid compositions.

Dishing

In order to evaluate dishing, a wafer chip of a square with a side of
5 20 mm was cut out from a wafer (commercially available from SKW,
"SKW6-2", size: 200 mm) with copper damascene interconnection patterns.
Five wafer chips were fixed on an adhesive plate made of ceramic.
Thereafter, a copper film in the periphery of the copper interconnection
portion, the interconnection of which had a width of 150 μm , was removed,
10 with confirming the state under the above conditions. The substrate was
polished to a point where a barrier film appeared, and was further polished
with 20% of a time period required for polishing until this point, to provide
a sample for evaluating dishing. The dishing was evaluated by determining
a cross section profile of a copper interconnection portion, the
15 interconnection of which had a width of 150 μm by a surface roughness
tester (commercially available from (K.K.) Mitsutoyo, "SV-600").
Incidentally, when there were no recesses having sizes of 0.15 μm or more
in the cross section profile of the copper interconnection tested, it was
evaluated as absence of dishing, and when there were recesses having sizes
20 of 0.15 μm or more, it was evaluated as presence of dishing, which are
respectively denoted in Table 2 as "absence" or "presence."

Here, the etching speed "b" of the polishing liquid compositions used
for Comparative Examples I-1 to I-3 were as follows.

Comparative Example I-1: 80 $\text{\AA}/\text{min}$

25 Comparative Example I-2: 80 $\text{\AA}/\text{min}$

Comparative Example I-3: 200 Å/min

Table 1

No.	Hydroxyl Group-Containing Compound
a	$\text{n-C}_4\text{H}_9\text{CH(OH)CH}_2\text{OH}$
b	$\text{n-C}_4\text{H}_9\text{OCH}_2\text{CH(OH)CH}_2\text{OH}$
c	$\text{n-C}_6\text{H}_{13}\text{COOCH}_2\text{CH(OH)CH}_2\text{OH}$
d	$(\text{n-C}_3\text{H}_7)_2\text{NCH}_2\text{CH(OH)CH}_2\text{OH}$
e	$\text{n-C}_3\text{H}_7\text{OCOCH(OH)CH(OH)COO-n-C}_3\text{H}_7$

Table 2

Ex. No.	Hydroxyl Group-Containing Compound		Organic Acid		Hydrogen Peroxide	Abrasive
	Kind	Content (% by wt.)	Kind	Content (% by wt.)	Content (% by wt.)	Kind
I-1	a	1.0	Gluconic acid	2.0	2.0	Fumed Silica
I-2	b	1.0	Gluconic acid	2.0	2.0	Fumed Silica
I-3	c	1.0	Gluconic acid	2.0	2.0	Fumed Silica
I-4	d	1.0	Gluconic acid	2.0	2.0	Fumed Silica
I-5	e	1.0	Gluconic acid	2.0	2.0	Fumed Silica
I-6	a	1.0	Gluconic acid	2.0	2.0	Colloidal Silica
I-7	b	1.0	Gluconic acid	2.0	2.0	Colloidal Silica
I-8	c	1.0	Gluconic acid	2.0	2.0	Colloidal Silica
I-9	a	0.5	Gluconic acid	2.0	2.0	Fumed Silica
I-10	a	2.0	Gluconic acid	2.0	2.0	Fumed Silica
I-11	b	1.0	—	—	2.0	Fumed Silica
I-12	b	1.0	Gluconic acid	2.0	—	Colloidal Silica
I-13	a	1.0	Glycolic acid	2.0	2.0	Colloidal Silica
I-14	b	1.0	Glycolic acid	2.0	2.0	Colloidal Silica
Comp. Ex. No.						
I-1	—	—	Gluconic acid	2.0	2.0	Fumed Silica
I-2	—	—	Gluconic acid	2.0	2.0	Colloidal Silica
I-3	—	—	Glycolic acid	2.0	2.0	Colloidal Silica
I-4	Benzo-triazole	1.0	Gluconic acid	2.0	2.0	Fumed Silica

— Continued —

- Continued -

Ex. No.	Evaluation of Properties						Dishing
	Relative Polishing Speed			Relative Etching Speed			
	Based on C.Ex.I-1	Based on C.Ex.I-2	Based on C.Ex.I-3	Based on C.Ex.I-1	Based on C.Ex.I-2	Based on C.Ex.I-3	
I-1	1.0	—	—	0.1 or less	—	—	Absence
I-2	1.0	—	—	0.1 or less	—	—	Absence
I-3	1.0	—	—	0.15	—	—	Absence
I-4	1.1	—	—	0.15	—	—	Absence
I-5	1.1	—	—	0.2	—	—	Absence
I-6	—	1.0	—	—	0.1 or less	—	Absence
I-7	—	1.0	—	—	0.1 or less	—	Absence
I-8	—	1.1	—	—	0.15	—	Absence
I-9	1.1	—	—	0.1 or less	—	—	Absence
I-10	1.0	—	—	0.1 or less	—	—	Absence
I-11	0.7	—	—	0.1 or less	—	—	Absence
I-12	—	0.7	—	—	0.1 or less	—	Absence
I-13	—	—	0.9	—	—	0.1 or less	Absence
I-14	—	—	0.9	—	—	0.1 or less	Absence
Comp. Ex. No.							
I-1	1.0	—	—	1.0	—	—	Presence
I-2	—	1.0	—	—	1.0	—	Presence
I-3	—	—	1.0	—	—	1.0	Presence
I-4	0.1	—	—	0.1 or less	—	—	*

Note *: The polishing speed was too slow to be evaluated.

It is found from the results of Tables 1 and 2 that all of the polishing liquid compositions of Examples I-1 to I-14 where the hydroxyl group-containing compound is formulated in the polishing liquid composition suppressed the etching speed and the dishing is not generated without substantially lowering the polishing speed, as compared to the polishing liquid compositions of Comparative Examples I-1 to I-3 where the hydroxyl group-containing compound is not formulated.

In addition, it is found that the polishing liquid composition of Comparative Example I-4 where benzotriazole acting to suppressing etching was used in place of the hydroxyl group-containing compound, the polishing speed is extremely low.

Also, it is found that by using the hydroxyl group-containing compound, the etching agent, and the oxidizing agent, a higher polishing speed can be realized, and the dishing can be prevented.

Examples II-1 to II-12 and Comparative Examples II-1 to II-12

Each of the etching agents shown in Tables 3 and 4, and hydrogen peroxide were mixed with a given amount of water, so as to have the proportion shown in Tables 3 and 4. Further, 5% by weight portion of the abrasive shown in Tables 3 and 4 were mixed with stirring, and thereafter an aqueous ammonia was added so as to have a pH of the liquid mixture of 7.5 to 8. Separately from above, an aqueous ammonia was previously added to an aliphatic carboxylic acid having 7 to 24 carbon atoms to adjust the pH to 7.5 to 8, whereby preparing an aqueous solution of the aliphatic carboxylic acid or its salt. The aqueous solution of the aliphatic carboxylic

acid or its salt was mixed with the above liquid mixture with stirring, so as to have the proportion shown in Tables 3 and 4. Thereafter, a pH of the liquid mixture was adjusted to a value shown in Tables 3 and 4, to give each polishing liquid composition. Incidentally, each of the used abrasives was fumed silica (primary particle size: 50 nm) and colloidal silica (primary particle size: 100 nm). In addition, a rolled copper plate of a diameter of 50 mm and a plate thickness of 1 mm was polished under the same conditions as above by single-sided polishing machine.

In addition, the properties of the polishing liquid composition such as relative polishing speed, relative etching speed, dishing of the surface to be polished, and surface condition of copper were evaluated in accordance with the following methods. The results thereof are shown in Tables 3 and 4.

Relative Polishing Speed

The relative polishing speed is a value obtained by dividing the polishing speed of a polishing liquid composition by a polishing speed of a comparative example where the kinds and the amounts of an abrasive, an oxidizing agent and an etching agent were the same as the polishing liquid composition. The polishing speed was obtained by polishing a rolled copper plate of a diameter of 50 mm and a plate thickness of 1 mm under the polishing conditions mentioned above, determining a change in the thickness before and after polishing, and dividing the resultant value by a polishing time. Incidentally, the thickness of the copper plate was measured by using a high-precision digital analyzer "MINIAX" commercially available from K. K. Tokyo Seimitsu. Here, each of the

relative polishing speeds for Examples II-1 to II-6 was calculated based on Comparative Example II-1; the relative polishing speed for Example II-7 was calculated based on Comparative Example II-2; the relative polishing speed for Example II-8 was calculated based on Comparative Example II-3; the relative polishing speed for Example II-9 was calculated based on Comparative Example II-4; the relative polishing speed for Example II-10 was calculated based on Comparative Example II-5; the relative polishing speed for Example II-11 was calculated based on Comparative Example II-11; the relative polishing speed for Example II-12 was calculated based on Comparative Example II-12; the relative polishing speed for Comparative Example II-9 was calculated based on Comparative Example II-1; and the relative polishing speed for Comparative Example II-10 was calculated based on Comparative Example II-2. Incidentally, apart from the above, each of the relative polishing speeds for Comparative Examples II-6 to II-8 was calculated based on Comparative Example II-1.

Relative Etching Speed

The relative etching speed is a value obtained by dividing an etching speed of the polishing liquid composition comprising the above aliphatic carboxylic acid and/or its salt by an etching speed "c" of a polishing liquid composition not containing the above aliphatic carboxylic acid and/or its salt but having the same kinds and the amounts of an abrasive, an oxidizing agent and an etching agent as the polishing liquid composition. Here, each of the relative etching speeds for Examples II-1 to II-6 was calculated based on Comparative Example II-1; the relative etching speed for Example

II-7 was calculated based on Comparative Example II-2; the relative etching speed for Example II-8 was calculated based on Comparative Example II-3; the relative etching speed for Example II-9 was calculated based on Comparative Example II-4; the relative etching speed for Example II-10 was calculated based on Comparative Example II-5; the relative etching speed for Example II-11 was calculated based on Comparative Example II-11; the relative etching speed for Example II-12 was calculated based on Comparative Example II-12; the relative etching speed for Comparative Example II-9 was calculated based on Comparative Example II-1; and the relative etching speed for Comparative Example II-10 was calculated based on Comparative Example II-2. Incidentally, apart from the above, each of the relative etching speeds for Comparative Examples II-6 to II-8 was calculated based on Comparative Example II-1. Incidentally, each the etching speeds for each the polishing liquid compositions of Examples II-1 to II-12 and Comparative Examples II-7 to II-8 was a value measured under the same conditions as the above etching test C described above, except for using these polishing liquid compositions.

Dishing

Dishing was evaluated in the same manner as above. Incidentally, when there were no recesses having sizes of 0.15 μm or more in the cross section profile of the copper interconnection tested, it was evaluated as absence of dishing, and when there were recesses having sizes of 0.15 μm or more, it was evaluated as presence of dishing, which are respectively denoted in Tables 3 and 4 as "absence" or "presence."

Copper Surface Condition

The surface condition of the copper film at the copper interconnection portion, the interconnection of which has a width of 150 μm on the surface of wafer chip used in the evaluation of the dishing was observed by optical microscope, to confirm the presence or absence of roughening.

Here, the etching speed "c" of the polishing liquid compositions used for Comparative Examples II-1 to II-6 were as follows.

Comparative Example II-1: 50 $\text{\AA}/\text{min}$

Comparative Example II-2: 100 $\text{\AA}/\text{min}$

Comparative Example II-3: 200 $\text{\AA}/\text{min}$

Comparative Example II-4: 600 $\text{\AA}/\text{min}$

Comparative Example II-5: 50 $\text{\AA}/\text{min}$

Comparative Example II-6: 100 $\text{\AA}/\text{min}$

Table 3

Ex. No.	Aliphatic Carboxylic Acid and/or Its Salt		Etching Agent		Hydrogen Peroxide	Abrasive	pH
	Kind	Content	Kind	Content		Kind	
II-1	Octanoic acid	0.6 ¹⁾	Glycolic acid	2.0 ¹⁾	4.0 ¹⁾	Colloidal Silica	7.6
II-2	Nonanoic acid	0.3	Glycolic acid	2.0	4.0	Colloidal Silica	7.7
II-3	Heptanoic acid	1.2	Glycolic acid	2.0	4.0	Colloidal Silica	7.6
II-4	Decanoic acid	0.3	Glycolic acid	2.0	4.0	Colloidal Silica	7.7
II-5	Oleic acid	0.3	Glycolic acid	2.0	4.0	Colloidal Silica	8.0
II-6	Isooctanoic acid ²⁾	1.5	Glycolic acid	2.0	4.0	Colloidal Silica	7.7
II-7	Octanoic acid	1.2	Citric acid	2.0	2.0	Fumed Silica	7.6
II-8	Octanoic acid	1.0	Phthalic acid	2.0	2.0	Fumed Silica	7.9
II-9	Octanoic acid	1.5	Aminotri-(methylene-phosphonic acid)	2.0	2.0	Fumed Silica	7.6
II-10	Octanoic acid	0.4	Glycolic acid	2.0	—	Colloidal Silica	7.6
II-11	Octanoic acid	1.0	Hydrochloric acid	2.0	2.0	Colloidal Silica	7.7
II-12	Octanoic acid	0.5	Sulfuric acid	2.0	2.0	Colloidal Silica	7.9

— Continued —

100304231_011002

- Continued -

Ex. No.	Evaluation for Properties				
	Relative Polishing Speed	Relative Etching Speed	Comp. Ex. To Which Evaluation Was Based	Dishing	Copper Surface Condition
II-1	1.0	0.1 or less	Comp. Ex. II-1	Absence	No Roughening
II-2	1.0	0.1 or less	Comp. Ex. II-1	Absence	No Roughening
II-3	1.0	0.1 or less	Comp. Ex. II-1	Absence	No Roughening
II-4	0.9	0.1 or less	Comp. Ex. II-1	Absence	No Roughening
II-5	0.9	0.1 or less	Comp. Ex. II-1	Absence	No Roughening
II-6	1.0	0.1 or less	Comp. Ex. II-1	Absence	No Roughening
II-7	1.0	0.1 or less	Comp. Ex. II-2	Absence	No Roughening
II-8	0.9	0.1 or less	Comp. Ex. II-3	Absence	No Roughening
II-9	0.9	0.1 or less	Comp. Ex. II-4	Absence	No Roughening
II-10	0.9	0.1 or less	Comp. Ex. II-5	Absence	No Roughening
II-11	0.9	0.1 or less	Comp. Ex. II-11	Absence	No Roughening
II-12	0.9	0.1 or less	Comp. Ex. II-12	Absence	No Roughening

Note 1): % by weight

Note 2): Secanoic C8 acid (trade name, commercially available
from Exxon Chemicals K.K.)

10030424 011002

Table 4

Comp. Ex. No.	Aliphatic Carboxylic Acid and/or Its Salt		Etching Agent		Hydrogen Peroxide	Abrasive	pH
	Kind	Content	Kind	Content		Kind	
II-1	—	—	Glycolic acid	2.0 ¹⁾	4.0 ¹⁾	Colloidal Silica	7.6
II-2	—	—	Citric acid	2.0	2.0	Fumed Silica	7.6
II-3	—	—	Phthalic acid	2.0	2.0	Fumed Silica	7.9
II-4	—	—	Aminotri- (methylene- phosphonic acid)	2.0	2.0	Fumed Silica	7.6
II-5	—	—	Glycolic acid	2.0	—	Colloidal Silica	7.6
II-6	—	—	Glycolic acid Citric acid	1.0 1.0	4.0	Colloidal Silica	7.6
II-7	Octanoic acid Heptanoic acid	0.5 ¹⁾ 0.5	—	—	4.0	Colloidal Silica	7.8
II-8	Oleic acid	0.5	—	—	4.0	Colloidal Silica	7.6
II-9	Benzo- triazole	0.3	Glycolic acid	2.0	4.0	Colloidal Silica	7.6
II-10	Ammonium Poly- acrylate	1.0	Citric acid	2.0	2.0	Fumed Silica	7.6
II-11	—	—	Hydrochloric acid	2.0	2.0	Colloidal Silica	7.7
II-12	—	—	Sulfuric acid	2.0	2.0	Colloidal Silica	7.9

— Continued —

– Continued –

Comp. Ex. No.	Evaluation for Properties				
	Relative Polishing Speed	Relative Etching Speed	Comp. Ex. To Which Evaluation Was Based	Dishing	Copper Surface Condition
II-1	1.0	1.0	–	Presence	No Roughening
II-2	1.0	1.0	–	Presence	No Roughening
II-3	1.0	1.0	–	Presence	No Roughening
II-4	1.0	1.0	–	Presence	No Roughening
II-5	1.0	1.0	–	Presence	No Roughening
II-6	1.3	1.5	Comp. Ex. II-1	Presence	No Roughening
II-7	0.1 or less	0.1 or less	Comp. Ex. II-1	The polishing speed was too slow to be evaluated.	
II-8	0.1 or less	0.1 or less	Comp. Ex. II-1		
II-9	0.1 or less	0.1 or less	Comp. Ex. II-1		
II-10	1.2	0.1 or less	Comp. Ex. II-2	Absence	Presence of Roughening
II-11	1.0	1.0	–	Presence	No Roughening
II-12	1.0	1.0	–	Presence	No Roughening

Note 1): % by weight

100304231_011002

It is found from the results of Tables 3 and 4 that all of the polishing liquid compositions of Examples II-1 to II-12 where the aliphatic carboxylic acid having 7 to 24 carbon atoms is formulated in the polishing liquid composition suppressed the etching speed and the dishing is not generated without substantially lowering the polishing speed, as compared to the polishing liquid compositions of Comparative Examples II-1 to II-5, II-11 and II-12 where the aliphatic carboxylic acid is not formulated.

In addition, the polishing liquid composition of Comparative Example II-6 where two kinds of aliphatic carboxylic acids having 6 or less carbon atoms are used in combination without using the aliphatic carboxylic acid having 7 to 24 carbon atoms has high etching speed and the dishing generated.

In addition, it is found that all of the polishing liquid composition of Comparative Example II-7 where two kinds of the aliphatic carboxylic acids having 7 to 24 carbon atoms are used in combination; the polishing liquid composition of Comparative Example II-8 where the organic acid capable of forming an aqueous salt with copper is not formulated; and the polishing liquid composition of Comparative Example II-9 where benzotriazole for suppressing etching is formulated have extremely low polishing speeds, and that the polishing liquid composition of Comparative Example II-10 where ammonium polyacrylate is formulated has copper surface with roughening generated.

Therefore, an even higher polishing speed can be realized by using the aliphatic carboxylic acid having 7 to 24 carbon atoms in combination with an etching agent, and the dishing can be prevented.

Especially, from the viewpoint of keeping the low-foamability during polishing, it is more preferable that the aliphatic carboxylic acid having 7 to 24 carbon atoms is heptanoic acid, octanoic acid and nonanoic acid.

5 Examples III-1 to III-8 and Comparative Examples III-1 to III-5

Each of the etching agents shown in Table 5, and the amine compound were mixed with a given amount of water, so as to have the proportion shown in Table 5, and thereafter an aqueous ammonia was added so as to have a pH of the liquid mixture of 6 to 8. Further, hydrogen
10 peroxide was added so as to have a proportion shown in Table 5. Further, 5% by weight portion of an abrasive shown in Table 5 was mixed with stirring, and thereafter a pH of the liquid mixture was adjusted to the value shown in Table 5, to give a polishing liquid composition. Incidentally, each of the used abrasives was fumed silica (primary particle size: 50 nm) and
15 colloidal silica (primary particle size: 100 nm). In addition, a rolled copper plate of a diameter of 50 mm and a plate thickness of 1 mm was polished under the same conditions as above by single-sided polishing machine.

In addition, the properties of the polishing liquid composition such as relative polishing speed, relative etching speed, and dishing of the surface to
20 be polished were evaluated in accordance with the following methods. The results thereof are shown in Table 5.

Relative Polishing Speed

The relative polishing speed is a value obtained by dividing the
25 polishing speed of a polishing liquid composition by a polishing speed of a

comparative example where the kinds and the amounts of an abrasive, an oxidizing agent and an etching agent were the same as the polishing liquid composition. The polishing speed was obtained by polishing a rolled copper plate of a diameter of 50 mm and a plate thickness of 1 mm, determining a change in the thickness before and after polishing, and dividing the resultant value by a polishing time. Incidentally, the thickness of the copper plate was measured by using a high-precision digital analyzer "MINIAX" commercially available from K. K. Tokyo Seimitsu. Here, each of the relative polishing speeds for Examples III-1, III-2, III-4, III-5, and III-7 was calculated based on Comparative Example III-1; the relative polishing speed for Example III-6 was calculated based on Comparative Example III-2; the relative polishing speed for Example III-3 was calculated based on Comparative Example III-3; and the relative polishing speed for Example III-8 was calculated based on Comparative Example III-4. Incidentally, apart from the above, the relative polishing speed for Comparative Example III-5 was calculated based on Comparative Example III-1.

Relative Etching Speed

The relative etching speed is a value obtained by dividing an etching speed of the polishing liquid composition comprising the above amine compound and/or its salt by an etching speed "c" of a polishing liquid composition not containing the above amine compound and/or its salt but having the same kinds and the amounts of an abrasive, an oxidizing agent and an etching agent as the polishing liquid composition. Here, each of the

relative etching speeds for Examples III-1, III-2, III-4, III-5, and III-7 was calculated based on Comparative Example III-1; the relative etching speed for Example III-6 was calculated based on Comparative Example III-2; the relative etching speed for Example III-3 was calculated based on

5 Comparative Example III-3; and the relative etching speed for Example III-8 was calculated based on Comparative Example III-4. Incidentally, apart from the above, the relative etching speed for Comparative Example III-5 was calculated based on Comparative Example III-1.

10 Incidentally, each the etching speeds for each the polishing liquid compositions of Examples III-1 to III-8 and Comparative Example III-5 was a value measured under the same conditions as the above etching test C described above, except for using these polishing liquid compositions.

Dishing

15 Dishing was evaluated in the same manner as above. Incidentally, when there were no recesses having sizes of 0.15 μm or more in the cross section profile of the copper interconnection tested, it was evaluated as absence of dishing, and when there were recesses having sizes of 0.15 μm or more, it was evaluated as presence of dishing, which are respectively
20 denoted in Table 5 as "absence" or "presence."

Here, the etching speed "c" of the polishing liquid compositions used for Comparative Examples III-1 to III-4 were as follows.

Comparative Example III-1: 50 $\text{\AA}/\text{min}$

Comparative Example III-2: 100 $\text{\AA}/\text{min}$

25 Comparative Example III-3: 50 $\text{\AA}/\text{min}$

Comparative Example III-4: 50 Å/min

Table 5

Ex. No.	Amine Compound		Etching Agent		Hydrogen Peroxide	Abrasive Kind	pH
	Kind	Content (% by wt.)	Kind	Content (% by wt.)	Content (% by wt.)		
III-1	Octylamine	0.8	Glycolic acid	2.0	4.0	Colloidal Silica	7.6
III-2	Nonylamine	0.5	Glycolic acid	2.0	4.0	Colloidal Silica	7.6
III-3	Decylamine	1.0	Hydrochloric acid	2.0	2.0	Colloidal Silica	7.7
III-4	Oleylamine	0.4	Glycolic acid	2.0	4.0	Colloidal Silica	7.6
III-5	Dimethyl-dodecyl-amine	0.5	Glycolic acid	2.0	4.0	Colloidal Silica	7.6
III-6	Dimethyl-dodecyl-amine	0.8	Citric acid	2.0	2.0	Fumed Silica	7.6
III-7	Dodecyl-diethanol-amine	0.8	Glycolic acid	2.0	4.0	Colloidal Silica	7.6
III-8	Octylamine	0.5	Glycolic acid	2.0	—	Colloidal Silica	7.6
Comp. Ex. No.							
III-1	—	—	Glycolic acid	2.0	4.0	Colloidal Silica	7.6
III-2	—	—	Citric acid	2.0	2.0	Fumed Silica	7.6
III-3	—	—	Hydrochloric acid	2.0	2.0	Colloidal Silica	7.7
III-4	—	—	Glycolic acid	2.0	—	Colloidal Silica	7.6
III-5	Benzotriazole	0.3	Glycolic acid	2.0	4.0	Colloidal Silica	7.6

— Continued —

– Continued –

Ex. No.	Evaluation for Properties			
	Relative Polishing Speed	Relative Etching Speed	Comp. Ex. To Which Evaluation Was Based	Dishing
III-1	1.0	0.1 or less	Comp. Ex. III-1	Absence
III-2	0.9	0.1 or less	Comp. Ex. III-1	Absence
III-3	1.0	0.1 or less	Comp. Ex. III-3	Absence
III-4	0.9	0.1 or less	Comp. Ex. III-1	Absence
III-5	0.9	0.1 or less	Comp. Ex. III-1	Absence
III-6	0.9	0.1 or less	Comp. Ex. III-2	Absence
III-7	0.9	0.1 or less	Comp. Ex. III-1	Absence
III-8	0.9	0.1 or less	Comp. Ex. III-4	Absence
Comp. Ex. No.				
III-1	1.0	1.0	–	Presence
III-2	1.0	1.0	–	Presence
III-3	1.0	1.0	–	Presence
III-4	1.0	1.0	–	Presence
III-5	0.1 or less	0.1 or less	Comp. Ex. III-1	*

Note *: The polishing speed was too slow to be evaluated.

5 It is found from the results of Table 5 that all of the polishing liquid compositions of Examples III-1 to III-8 where the amine compound is formulated in the polishing liquid composition suppressed the etching speed and the dishing is not generated without substantially lowering the polishing speed, as compared to the polishing liquid compositions of Comparative Examples III-1 to III-4 where the amine compound is not formulated.

10 In addition, it is found that the polishing liquid composition of Comparative Example III-5 where benzotriazole acting to suppressing etching was used in place of the amine compound, the polishing speed is extremely low.

15

Therefore, it is found that an even higher polishing speed can be realized by using the amine compound in combination with an etching agent, and the dishing can be prevented.

Especially, from the viewpoint of keeping the low-foamability during polishing, it is more preferable that the amine compound is heptylamine, 5 octylamine and nonylamine.

Since the polishing liquid composition of the present invention is used for polishing a surface to be polished comprising an insulating layer and a metal layer, there are exhibited such effects that the polishing speed of the metal film is maintained, that the etching speed is suppressed, and defects 10 such as dishing in the interconnection metal layer is not generated.

10030424 011002

ART 34 AMDT

63

CLAIMS

1. A polishing liquid composition for polishing a surface to be polished comprising an insulating layer and a metal layer, the polishing liquid composition comprising a compound having a structure in which each of two or more adjacent carbon atoms has a hydroxyl group in a molecule, and water, wherein the compound having a structure in which each of two or more adjacent carbon atoms has a hydroxyl group in a molecule is represented by the formula (I):



wherein R^1 is a hydrocarbon group having 1 to 24 carbon atoms; X is a group represented by $(CH_2)_m$, wherein m is 1, oxygen atom, sulfur atom, COO group, OCO group, a group represented by NR^2 or $O(R^2O)P(O)O$, wherein R^2 is hydrogen atom or a hydrocarbon group having 1 to 24 carbon atoms; q is 0 or 1; and n is an integer of 1 to 4.

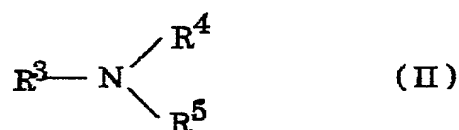
2. The polishing liquid composition according to claim 1, further comprising an organic acid.

3. The polishing liquid composition according to claim 2, wherein the organic acid is an etching agent.

4. The polishing liquid composition according to claim 1, further comprising an etching agent comprising an inorganic acid.

5. A polishing liquid composition for polishing a surface to be polished comprising an insulating layer and a metal layer, the polishing liquid composition comprising an aliphatic carboxylic acid having 7 to 24 carbon atoms and/or a salt thereof, an etching agent comprising an organic acid, and water.

6. A polishing liquid composition for polishing a surface to be polished comprising an insulating layer and a metal layer, the polishing liquid composition comprising an amine compound represented by the following general formula (II):



wherein R³ is a linear or branched alkyl group having 4 to 18 carbon atoms, a linear or branched alkenyl group having 4 to 18 carbon atoms, an aryl group having 6 to 18 carbon atoms, and an aralkyl group having 7 to 18 carbon atoms; each of R⁴ and R⁵, which may be identical or different, is hydrogen atom, a linear alkyl group having 1 to 8 carbon atoms or a branched alkyl group having 3 to 8 carbon atoms, or a group represented by H-(OR⁶)_Z, wherein R⁶ is a linear alkylene group having 1 to 3 carbon atoms, or a branched alkylene group having 3 carbon atoms; and Z is a number of 1 to 20, and/or a salt thereof, an etching agent, an oxidizing agent, and water.

7. The polishing liquid composition according to any one of claims 1 to 5,
further comprising an oxidizing agent.

8. The polishing liquid composition according to any one of claims 1 to 7,
further comprising an abrasive.

9. A process for polishing a semiconductor substrate, comprising
polishing a surface to be polished comprising an insulating layer and a
metal layer using the polishing liquid composition of any one of claims 1 to 8,
thereby smoothening the semiconductor substrate.

10. A process for manufacturing a semiconductor substrate comprising
polishing a surface to be polished comprising an insulating layer and a
metal layer using the polishing liquid composition of any one of claims 1 to 8,
thereby smoothening the semiconductor substrate.

Declaration and Power of Attorney For Patent Application

特許出願宣言書及び委任状

Japanese Language Declaration

日本語宣言書

下記の氏名の発明者として、私は以下の通り宣言します。

私の住所、私書箱、国籍は下記の私の氏名の後に記載された通りです。

下記の名称の発明に関して請求範囲に記載され、特許出願している発明内容について、私が最初かつ唯一の発明者（下記の氏名が一つの場合）もしくは最初かつ共同発明者（下記の名称が複数の場合）であると信じています。

上記発明の明細書は、

- ☐ 本書に添付されています。
- ☐ ____月____日に提出され、米国出願番号または特許協定条約国際出願番号を____とし、
(該当する場合) ____に訂正されました。

私は、特許請求範囲を含む上記訂正後の明細書を検討し、内容を理解していることをここに表明します。

私は、連邦規則法典第37編第1条56項に定義されたとおり、特許資格の有無について重要な情報を開示する義務があることを認めます。

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled.

POLISHING LIQUID COMPOSITION

the specification of which

- ☐ is attached hereto.
- ☒ was filed on July 7, 2000
as United States Application Number or
PCT International Application Number
PCT/JP00/04571 and was amended on
____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56.

Japanese Language Declaration
(日本語宣言書)

私は、米国法典第35編119条 (a) - (d) 項又は365条 (b) 項に基づき下記の、米国以外の国の少なくとも一カ国を指定している特許協力条約365 (a) 項に基づく国際出願、又は外国での特許出願もしくは発明者証の出願についての外国優先権をここに主張するとともに、優先権を主張している、本出願の前に出願された特許または発明者証の外国出願を以下に、枠内をマークすることで、示しています。

Prior Foreign Application(s)

外国での先行出願

11-198263

(Number)
(番号)

2000-30477

(Number)
(番号)

Japan

(Country)
(国名)

Japan

(Country)
(国名)

私は、第35編米国法典119条 (e) 項に基づいて下記の米国特許出願規定に記載された権利をここに主張いたします。

(Application No.)
(出願番号)

(Filing Date)
(出願日)

私は、下記の米国法典第35編120条に基づいて下記の米国特許出願に記載された権利、又は米国を指定している特許協力条約365条 (c) に基づく権利をここに主張します。また、本出願の各請求範囲の内容が米国法典第35編112条第1項又は特許協力条約で規定された方法で先行する米国特許出願に開示されていない限り、その先行米国出願書提出日以降で本出願書の日本国内または特許協力条約国際提出日までの期間中に入手された、連邦規則法典第37編1条56項で定義された特許資格の有無に関する重要な情報について開示義務があることを認識しています。

(Application No.)
(出願番号)

(Filing Date)
(出願日)

(Application No.)
(出願番号)

(Filing Date)
(出願日)

私は、私自信の知識に基づいて本宣言書中で私が行なう表明が真実であり、かつ私の入手した情報と私の信じるところに基づく表明が全て真実であると信じていること、さらに故意になされた虚偽の表明及びそれと同等の行為は米国法典第18編第1001条に基づき、罰金または拘禁、もしくはその両方により処罰されること、そしてそのような故意による虚偽の声明を行えば、出願した、又は既に許可された特許の有効性が失われることを認識し、よってここに上記のごとく宣誓を致します。

I hereby claim foreign priority under Title 35, United States Code, Section 119 (a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or Section 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

Priority Claimed

優先権主張

13/07/1999

(Day/Month/Year Filed)
(出願年月日)

08/02/2000

(Day/Month/Year Filed)
(出願年月日)

☒ Yes ☐ No

はい いいえ

☒ Yes ☐ No

はい いいえ

I hereby claim the benefit under Title 35, United States Code, Section 119(e) of any United States provisional application(s) listed below.

(Application No.)
(出願番号)

(Filing Date)
(出願日)

I hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s), or Section 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code Section 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of application.

(Status: Patented, Pending, Abandoned)
(現況: 特許許可済、係属中、放棄済)

(Status: Patented, Pending, Abandoned)
(現況: 特許許可済、係属中、放棄済)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Japanese Language Declaration
(日本語宣言書)

委任状：私は下記の発明者として、本出願に関する一切の手続きを米特許商標局に対して遂行する弁理士または代理人として、下記の者を指名いたします。
(弁理士、または代理人の指名及び登録番号を明記のこと)

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith: (list name and registration number)



022850

書類送付先

Send Correspondence to:



022850

直接電話連絡先：(名前及び電話番号)

Direct Telephone Calls to: (name and telephone number)
(703) 413-3000

単独発明者または第一の共同発明者の氏名	Full name of sole or first joint inventor Yasuhiro YONEDA
発明者の署名 日付	Inventor's signature Date Yasuhiro Yoneda December 18, 2001
住所	Residence Wakayama-shi, Wakayama 640-8580 Japan
国籍	Citizenship Japan
郵便の宛先	Post Office Address c/o Kao Corporation, Research Laboratories, 1334, Minato, Wakayama-shi, Wakayama 640-8580 Japan
第二の共同発明者の氏名	Full name of second joint inventor, if any Ryoichi HASHIMOTO
第二の共同発明者の署名 日付	Second joint inventor's signature Date Ryoichi Hashimoto December 18, 2001
住所	Residence Wakayama-shi, Wakayama 640-8580 Japan
国籍	Citizenship Japan
郵便の宛先	Post Office Address c/o Kao Corporation, Research Laboratories, 1334, Minato, Wakayama-shi, Wakayama 640-8580 Japan

(第三以降の共同発明者についても同様に記載し、署名すること)

(Supply similar information and signature for third and subsequent joint inventors.)

Japanese Language Declaration
(日本語宣言書)

第三の共同発明者の氏名	Full name of third joint inventor, if any Toshiya HAGIHARA
第三の共同発明者の署名 日付	Third joint Inventor's signature Date Toshiya Hagihara December 18, 2001
住所	Residence Wakayama-shi, Wakayama 640-8580 Japan
国籍	Citizenship Japan
郵便の宛先	Post Office Address c/o Kao Corporation, Research Laboratories,
	1334, Minato, Wakayama-shi, Wakayama 640-8580 Japan.

第四の共同発明者の氏名	Full name of fourth joint inventor, if any
第四の共同発明者の署名 日付	Fourth joint Inventor's signature Date
住所	Residence
国籍	Citizenship
郵便の宛先	Post Office Address

第五の共同発明者の氏名	Full name of fifth joint inventor, if any
第五の共同発明者の署名 日付	Fifth joint Inventor's signature Date
住所	Residence
国籍	Citizenship
郵便の宛先	Post Office Address

第六の共同発明者の氏名	Full name of sixth joint inventor, if any
第六の共同発明者の署名 日付	Sixth joint Inventor's signature Date
住所	Residence
国籍	Citizenship
郵便の宛先	Post Office Address

(第六またはそれ以降の共同発明者に対しても同様な情報および署名を提供すること。)

(Supply similar information and signature for third and subsequent joint inventors.)